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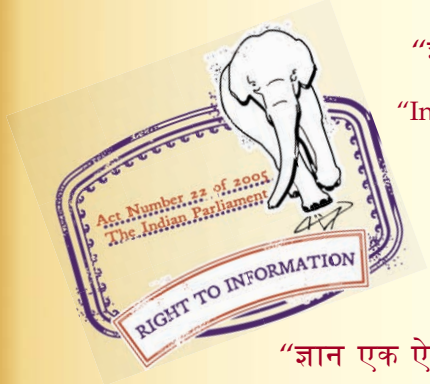
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IS 1164 (1986): Cocoa Powder [FAD 6: Stimulant Foods]

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SPECIFICATION FOR COCOA POWDER

(Third Revision)

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NEW DELHI 110002

Gr 5 *December* 1986

Indian Standard

SPECIFICATION FOR COCOA POWDER

(Third Revision)

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AMENDMENT NO. 1 JUNE 2006
TO
IS 1164 : 1986 SPECIFICATION FOR COCOA POWDER

(Third Revision)

(Page 12, *Appendix* F, *clause* **F-3.1**) — Substitute *'47l' for* '47.1'.

(FAD 6)

AMENDMENT NO. 2 DECEMBER 2009
TO
IS 1164 : 1986 SPECIFICATION FOR COCOA POWDER

(Third Revision)

[Page 6, clause 5.1, *SI No.* (e)] — Substitute 'Net quantity' *for* 'Net mass'.

[Page 6, *clause* 5.1, *SI No.* (g)] — Substitute the following for the existing:

'Any other requirements as given under the *Standards of Weights and Measures (Packaged Commodities) Rules,* 1977 and the *Prevention of Food Adulteration Act,* 1955 and the Rules.'

(FAD 6)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR COCOA POWDER

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Indian Standards Institution on 30 June 1986, after the draft finalized by the Stimulant Foods Sectional Committee had been approved by the Agricultural and Food Products Divison Council.

0.2 Since the second revison of this standard in 1980, the need for updating the standard in order to accommodate differences in requirements for alkalized and non-alkalized cocoa powders, has been felt. In this revision, microbial limits have also been specified in order to produce cocoa powder hygienically.

0.2.1 The third revison is brought out to update the standard. Also the nomenclature for types of cocoa powder has been revised. The alkalized types of high fat, and low fat cocoa powders have been included, while break-fast cocoa powder and solubilized cocoa powder, being trade terminology have been deleted.

0.3 Cocoa powder is the partially defatted product derived from the Cocoa bean, the seed of *Theobroma cacao* L. The beans obtained from ripe pods are well fermented, dried, cleaned, roasted, cracked and freed from shell to obtain the nib. The nib is then finely ground in a cocoa liquor mill, when owing to friction heat, the fat, cocoa butter is liberated and the fluid cocoa mass obtained. Cocoa butter is subsequently extracted from this fluid mass in a hydraulic press. The resulting cocoa cake, is then cooled, broken and finely pulverized to obtain cocoa powder. Alkalized cocoa powder is produced by treatment of cocoa nibs or cocoa mass with solutions or suspensions of alkali, usually in the form of carbonates or bicarbonates of Soduim & Potassium, Calcium hydroxide or Ammonium carbonate. This treatment darkens the colour modifies the flavour and also improves the suspensibility (dispersibility) of cocoa powder in the beverage. The alkalization treatment is followed by drying to remove the excess moisture from the product.

0.4 While formulating this standard, due consideration has been given to the relevant rules issued by the Government of India under the *Prevention*

of Food Adulteration Act (PFA), 1954. This standard is, however subject to the restrictions imposed under that Act and the Rules framed thereunder; wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with **IS** : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 The standard prescribes the requirements and methods of test for cocoa powder.

2. TYPES

2.1 Cocoa powder shall be of the following types:

a) High fat cocoa powder, and

b) Low fat cocoa powder.

3. REQUIREMENTS

3.1 Description — Cocoa powder shall be the material obtained by mechanical transformation into powder form of cocoa press cake resulting from the partial removal of fat from the ground nib of well-fermented sound and wholesome roasted beans of *Theobroma cacao* L. It shall be in the form of a free flowing powder having a colour, taste and flavour characteristic of a good cocoa powder. It shall be free from direct or other extraneous matters, rancidity or other off odours, insect infestation, mould growth, adulterants or other harmful ingredients. It shall not contain added colouring matter.

3.2 The extracted fat from cocoa powder shall conform to IS : 1263-1986-†.

3.3 Particle Size — When tested by the method prescribed in Appendix A, not less than 99 percent of the material shall pass through 150-micron IS Sieve [*see IS* : 460 (Part 1)-1978‡, IS : 460 (Part 2)-1978‡ and IS : 460 (Part 3)-1978‡].

•Rules for rounding off numerical values (*revised*).
†Specifiication for cocoa butter.
‡Specification for test sieves:
Part 1 Wire cloth test sieves (*second revision*).
Part 2 Perforated plate test sieves (*second revision*).
Part 3 Methods of examination of test sieves (*second revision*).

3.4 The material shall also comply with requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR COCOA POWDER

SL NO	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST, REF TO	
		High Fat Cocoa Powder	Low Fat Cocoa Powder	Appendix of this Standard	Other Related Indian Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, percent by mass, *Max*	7·0	7·0	B	—
ii)	Cocoa butter (on moisture free basis), percent by mass, *Min*	20·0	10·0	C	—
iii)	Total ash (on moisture & fat free basis), percent by mass, *Max*	8·0	8·0	D	—
iv)	Acid insoluble ash (on moisture and fat free basis), percent by mass, *Max*	0·20	0·20	E	—
v)	Alkalinity of total ash as K2O (on moisture and fat free basis), percent by mass, *Max*	5·0	5·0	F	—
vi)	Crude fibre (on moisture and fat free basis), percent by mass, *Max*	7·0	7·0	G	—
vii)	*Staphylococcus aureus*	Absent	Absent	—	IS : 5887 (Part 2) - 1976*
viii)	*Salmonella*	Absent	Absent	—	IS : 5887 (Part 3) - 1976*
ix)	*E. coli* per 10 g	Absent	Absent	—	IS : 5887 (Part 1) - 1976*
x)	Yeast and mould count, per gm, *Max*	100	100	—	IS : 5403 - 1969†

*Methods for detection of bacteria responsible for food poisoning:
Part 2 Isolation, identification and enumeration of staphylococcus aureus and faecal streptococci (*first revision*).
Part 3 Isolation, and identification of salmonella and shigella (*first revision*).
Part 1 Isolation, identification and enumeration of escherichia coli (*first revision*).

†Method for yeast and mould count of foodstuffs.

3.5 Optional Treatment and Ingredients — The material may at any stage during its manufacture be treated with alkalizing agents, such as hydroxides, carbonates and bicarbonates of sodium, potassium, magnesium and aluminium only not exceeding 5 percent singly or in combination and expressed as anhydrous potassium carbonate on fat free basis. The maximum level of neutralizing agents used, namely, citric, tartaric or phosphoric acid shall be 0·5 percent in the final product. The material may contain flavouring agents, spices and common salt. It may also contain- emulsifiers permitted under the PFA Rules. For alkalized cocoa powder, (a) total ash (on moisture and fat free basis) percent by mass *Max,*14 percent, and (b) alkalinity of total ash as K_2O (on moisture and fat free basis, percent by mass, *Max 6* percent).

4. PACKING

4.1 Unless otherwise agreed to between the purchaser and the vendor, the material shall be packed in clean, sound and suitably lined-in containers and sealed airtight.

5. MARKING

5.1 The following particulars shall be marked or labelled legibly and indelibly on each container:

a) Name of the material.

b) Type,

c) Name and complete address of the manufacturer,

d) Batch or code number,

e) Net mass,

f) Date of manufacture, and

g) Any other particulars required under the Standards of *Weights and Measures (Packaged Commodities) Rules,* 1977.

5.2 The product may also be marked with Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau Of Indian Standards.

6. SAMPLING

6.1 The representative samples of the material shall be drawn according to the method prescribed in Appendix H.

7. TESTS

7.1 Tests shall be carried out as prescribed in appropriate appendices specified in 3.3 and in col 5 of Table 1.

7.2 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 3.3)

DETERMINATION OF PARTICLE SIZE

A-1. APPARATUS

A-1.1 Soxhlet Apparatus — sufficiently large to take a thimble containing 100 g of the material.

A-2. REAGENT

A-2.1 Petroleum Ether — boiling range 40 to 60 °C.

A-3. PROCEDURE

A-3.1 Place about 100 g of the material in the Soxhlet apparatus and extract with petroleum ether for one hour. At the end of this period, remove the fat-free material from the thimble and evaporate the petroleum ether completely on a steam-bath, and disintegrate any lumps that might have been formed. Weigh accurately about 25 g of this material and place it on an 150-micron IS Sieve. Shake in a suitable mechanical shaker for 2 minutes. Weigh the material that has passed through the sieve.

*Specification for water for general laboratory use (*second revision*).

A-4. CALCULATION

A-4.1 Material passing through sieve, percent by mass $\frac{100\ M_1}{M_2}$

where

M_1 = mass in g of the material that has passed through the sieve, and

M_2 = mass in g of fat-free material taken for the sieving.

APPENDIX B

*[**Table** 1, **Item** (i)]*

DETERMINATION OF MOISTURE

B-0. GENERAL

B-0.1 Two methods, namely, vacuum-oven method (*see* **B**-1) and air-oven method (*see* **B**-2), are included for determining the moisture content of the material. Anyone of these methods may be followed. The method followed shall be stated while submitting the test results.

B-1. VACUUM-OVEN METHOD

B-1.1 Procedure — Weigh accurately 10 g of the material in a tard weighing bottle having a diameter of about 40 mm and a height of about 25 mm. Distribute the material as evenly as possible over the bottom of the bottle by gently tapping. Place the bottle in a vacuum oven, remove the cover of the bottle and dry the material for six hours at 80 ± 1°C at a pressure not exceeding 5 mm of mercury. Allow the bottle to cool to room temperature and weigh.

B-1.2 Calculation — Moisture, percent by mass = $\frac{100\ (M - M_1)}{M}$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after drying.

B-2. AIR-OVEN METHOD

B-2. Procedure — Weigh accurately 2 g of the material in a tared petri dish. Distribute the material as evenly as possible and place it in an air-oven and dry the material at 100°C to a constant mass.

B-2.2 Calculation — Moisture, percent by mass $\frac{100\ (M - M_1)}{M}$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after drying.

APPENDIX C

[Table 1, *Item* (ii)]

DETERMINATION OF COCOA BUTTER

C-1. APPARATUS

C-1.1 Soxhlet Apparatus — with 250 ml flat bottom flask.

C-2. REAGENT

C-2.1 Petroleum Ether — redistilled below 60 to 80°C.

C-3. PROCEDURE

C-3.1 Weigh accurately 5 to 10 g of the moisture free material and transfer it to the fat extraction thimble of the Soxhlet apparatus. Extract the fat with petroleum ether for about 16 hours. Continue the extraction till at least 300 ml of petroleum ether have been circulated. Dry the fat (cocoa butter) at 70°C in a vacuum oven or at 100-110°C in an air oven till the difference in mass between two successive weighings is not more than 1 mg.

C-4. CALCULATION

C-4.1 Cocoa butter (on moisture free basis), percent by mass

$$= \frac{10\ 000\ m_1}{m_2\ (100 - M)}$$

where

m_1 = mass in g of fat,

m_2 = mass in g of moisture free material taken for the test, and

M = moisture, percent by mass, in the material (*see* B-2.1).

APPENDIX D

[Table 1, *Item* (iii)]

DETERMINATION OF TOTAL ASH

D-1. PROCEDURE

D-1.1 Weigh accurately 10 g of the material in a porcelain dish. Heat at 100°C until water is expelled and then heat slowly over a flame until swelling ceases. Ignite in a muffle furnace at 550°C until grey ash results. Cool in a desiccator and weigh. Repeat the process of igniting, cooling and weighing at half-hour intervals until the difference in mass between two successive weighings is less than 1 mg.

NOTE — Preserve the dish containing this ash for the determination of acid insoluble ash (*see* E-2.1).

D-2. CALCULATION

D-2.1 Total ash (on moisture and cocoa-butter free basis), percent by mass

$$= \frac{10\ 000\ m_1}{m_2\ [100 - (M + F)]}$$

where

m_x = mass in g of the ash;

m_2 = mass in g of the material taken for the test;

M = moisture, percent by mass, in the material (*see* B-2.1); and

F = fat (cocoa-butter), percent by mass in the material *(see* C-4.1).

APPENDIX E

[Table 1, *Item* (iv)]

DETERMINATION OF ACID INSOLUBLE ASH

E-1. REAGENT

E-1.1 Dilute Hydrochloric Acid — approximately 5 N, prepared from concentrated hydrochloric acid (**see** IS : 265-1962*).

E-2. PROCEDURE

E-2.1 To the ash contained in the dish (**see D-1.1)** add 25 ml of dilute hydrochloric acid, cover the dish with a watch glass and heat it on a boiling water bath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper till the washings are free from the acid. Return the filter paper and the residue to the dish. Keep in an electric air-oven maintained at 135 ± *2°C* for about 3 hours. Ignite in a muffle furnace at 550°C for one hour. Cool the dish in a desiccator and weigh. Repeat the process of igniting in a muffle furnace, cooling and weighing at half an hour intervals until the difference in mass between two successive weighings is less than one milligram. Note the lowest mass.

E-3. CALCULATION

E-3.1 Acid insoluble ash (on moisture and fat-free basis), percent by mass

$$= \frac{10\,000\ m_1}{m_2\,[\,100-(M+F)\,]}$$

where

m_1 = mass in g of the acid insoluble ash;

m_2 = mass in g of the material taken for the test:

M = moisture, percent by mass, in the material (***see*** **B-2.1**); and

F = fat (cocoa butter), percent by mass in the material *(**see** **C-4.1**).*

*Specification for hydrochloric acid (*revised).*

APPENDIX F

[Table 1, *Item* (v)]

DETERMINATION OF ALKALINITY OF ASH

F-l. REAGENTS

F-1.1 Dilate Hydrochloric Acid — approximately 0-1 N.

F-1.2 Standard Sodium Hydroxide — approximately 0·1 N, accurately standardized.

F-2. PROCEDURE

F-2.1 Weigh accurately about 2 g of the material, and ash as prescribed in **D-1.1**. Add a known excess of dilute hydrochloric acid and boil for 2 minutes. Cool and titrate the excess of acid against standard sodium hydroxide using bromocresol green as indicator, till the colour changes to green.

F-2.1.1 Titrate 10 ml of dilute hydrochloric acid against the standard sodium hydroxide using phenolphthalein as indicator, till the colour changes to pink.

F-3. CALCULATION

F-3.1 Alkalinity of ash (as K_aO) (on moisture and fat free basis), percent by mass

$$= \frac{47{\cdot}1\ N \left(\frac{(V_2\ V_1}{10} - V_3 \right)}{m\,[\,100 - (M + F)\,]}$$

where

N = normality of standard sodium hydroxide (*see* **F-1.2**);

V_2 — volume in ml of dilute hydrochloric acid added (*see* **F-2.1**);

$V1$ = volume in ml of standard sodium hydroxide corresponding to 10 ml of dilute hydrochloric acid;

v_3 = volume in ml of standard sodium hydroxide required for the excess of acid;

m = mass in g of the material taken for the test (*see* **F-2.1**);

M = moisture, percent by mass, in the material *(see* ***F-2.1***); and

F = fat (cocoa-butter), percent by mass, in the material *(see* **C-4.1**).

APPENDIX G

[Table 1, Item (vi)]

DETERMINATION OF CRUDE FIBRE

G-1. REAGENT

G-1.1 Ether — solvent grade (*see* IS : 336-1964*).

G-1.2 Ethyl Alcohol — *see* IS : 321-1964f.

G-1.3 Sodium Oxalate Solution — one percent (*m/v*).

G-1.4 Dilute Sulphuric Acid — 1 -25 percent (*mjv*), accurately prepared.

G-1.5 Sodium Hydroxide Solution— 125 percent *(mjv),* accurately prepared.

G-1.6 Asbestos — Gooch-grade, medium fibre, acid-washed and ignited.

G-2. PROCEDURE

G-2.1 Preparation of Moisture and Fat Free Material — Treat 7 g of the material in a centrifuge bottle twice with 100 ml of ether. Centrifuge and decant supernatant liquid after each addition of ether. Dry the residue in an air-oven at 100 ± 2°C and powder it with a flattened glass rod. Add 100 ml of water, centrifuge for 10 minutes and decant the aqueous layer. Repeat the washing with water twice. Then wash twice with 100 ml of alcohol and once with 100 ml ether, in the same manner as with water. Transfer the residue to a dish and dry in an air-oven at 105±1°C till the difference in mass between two successive weighings is not more than 1 mg. Use this for determination of crude fibre (*see* **G-2.2**).

*Specification for ether (*revised*).
†Specification for absolute alcohol (*revised*).

G-2.2 Weigh accurately about 2·5 g of the moisture and fat-free material *(see* **G-2.1**) and transfer to a litre flask. Take 200 ml of dilute sulphuric acid in a beaker and bring to boil. Transfer all the boiling sulphuric acid to the flask containing the fat-free material and immediately connect with a reflux water condenser. Heat, so that the contents of the flask begin to boil within 1 minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask out of contact with the acid. Continue boiling exactly for 30 minutes. Remove the flask and filter through fine linen (about 18 threads to a centimetre) held in a funnel. Wash with boiling water until the washings are no longer acid to litmus. Heat some quantity of sodium hydroxide solution to boiling under a reflux condenser. Wash the residue on the linen into the litre flask with 200 ml of the boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the filtering cloth. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of the ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of ethyl alcohol (95 percent by volume). Dry the Gooch crucible and contents at 105°C in an air-oven to constant mass. Cool and weigh. Incinerate the contents of the Gooch crucible in an electric muffle furnace at 600 ± 20°C until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

G-3. CALCULATION

G-3.1 Crude fibre (on moisture and fat free basis),

$$\text{percent by mass} = \frac{70\ (M_1 - M_2)}{M}$$

where

M_1 = mass in g of Gooch crucible and contents before ashing;

M_2 = mass in g of Gooch crucible containing asbestos and ash; and

M = mass in g of the moisture and fat free material taken for the (*see* **G-2.1**).

APPENDIX H

(Clause 6.1)

SAMPLING OF COCOA POWDER

H-1. GENERAL REQUIREMENTS OF SAMPLING

H-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

H-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

H-1.2 The sampling instrument, preferably a spoon or spatula, shall be clean and dry when used.

H-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

H-1.4 The samples shall be placed in clean and dry glass or tin containers. The sample containers shall be of such size that they are almost completely filled by the sample.

H-1.5 Each container shall be sealed airtight after filling and marked with full details of sampling, batch or code number, name of the manufacturer, sub-group number and other important particulars of the consignment and lot.

H-1.6 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and that they are protected from light.

H-2. SCALE OF SAMPLING

H-2.1 Lot — All the containers of the same size in a single consignment of material drawn from a single batch of manufacture shall constitute a lot.

H-2.2 Samples shall be tested for each lot separately for ascertaining conformity of the material to the requirements of this specification. The total number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2 and Table 3.

H-2.2.1 These containers shall be chosen at random from the lot and for this purpose a random number table as agreed to between the purchaser and the vendor shall be used. In case such a table is not available, the following procedure shall be adopted.

Starting from any container, count in one order as 1, 2, 3. . . . .etc, up to *r* and so on, where r is the integral part of *N/r* (N being the number of containers in the lot and *n* the number of containers to be selected). Every rth container so counted shall be withdrawn to constitute the sample.

H-3. TEST SAMPLES AND REFEREE SAMPLES

H-3.1 The sample containers of net contents less than 500 g selected according to H-2.2 and col 1 and 2 of Table 2 shall be equally divided at random into a number of groups specified in col 3 of Table 2. Each sample container of net content 500 g or more selected according to **H-2.2** and col 1 and 2 of Table 3 shall be treated as one group.

TABLE 2 SAMPLING OF CONTAINERS OF NET CONTENT LESS THAN 500 g

(Clauses H-2.2 *and* H-3.1)

NUMBER OF CONTAINERS IN THE LOT	TOTAL NUMBER OF CONTAINERS TO BE SELECTED	NUMBER OF GROUPS INTO WHICH SAMFLB CONTAINERS HAVE TO BE DIVIDED
(1)	(2)	(3)
Up to 50	*9*	1
51 to 300	18	2
301 to 500.	20	2
501 to 1 000	30	3
1 001 to 3 000	40	4
3 001 and above	50	5

TABLE 3 SAMPLING OF CONTAINERS OF NET CONTENT 500 g OR MORE

***(Clauses* H-2.2 *and* H-3.1)**

NUMBER OF CONTAINERS IN LOT	TOTAL NUMBER OF CONTAINERS TO BE SELECTED
Up to 50	**2**
51 to 300	3
301 to 500	4
501 to 1 000	5
1 001 and above	**6**

H-3.2 Preparation of Individual Samples — The contents of all the containers in a group shall be poured out and mixed thoroughly. About 360 g of the material shall be taken from this and divided into three equal parts. Each part, so obtained, shall be transferred to a sample container which shall be sealed airtight and labelled with the particulars given in **H-1.5**. The samples so obtained shall be divided into three sets in such a way that each set has a sample representing each group. One of these sets shall be marked for the purchaser, another for the vendor and the third for the referee.

H-3.3 Preparation of a Composite Sample — From the mixed material of each group remaining after taking the sample in **H-3.2,** equal quantities of the material shall be taken and mixed up together so as to form a composite sample representing the lot as a whole and weighing not less than 120 g. This composite sample shall be divided into three equal parts and transferred to clean, dry sample containers and labelled with all particulars given in **H-1.5**. One of these composite samples representing the lot as a whole shall be for the purchaser, another for the vendor and the third for the referee.

H-3.4 Referee Samples — Referee samples for a lot shall consist of a set of samples obtained in H-3.2 and a composite sample obtained in **H-3.3,** marked for this purpose, and shall bear the seals of the purchaser and the vendor. These shall be kept at a place and under conditions agreed to between the purchaser and the vendor.

H-4. NUMBER OF TESTS

H-4.1 The test for the description, particle size, and moisture shall be conducted on the individual samples as obtained in **H-3.2**.

H-4.2 The tests for cocoa-butter, total ash, acid insoluble ash, alkalinity and crude fibre shall be made on the composite sample as prepared under **H-3.3**.

H-5. CRITERIA FOR CONFORMITY

H-5.1 The lot shall be considered as conforming to the requirements of this specification if **H-5.1.1** and **H-5.1**.2 are satisfied.

H-5.1.1 The results of the tests conducted on the individual samples, for the characteristics specified in **H-4.1** shall satisfy the corresponding requirement as given in 3.

H-5.1.2 The results of the test conducted on the composite sample for the various characteristics mentioned in **H-4.2** shall satisfy the corresponding requirements as given in 3.

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